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NRL Report 6653

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A Computer Program for the Quantitative Interpretation of Mass Spectrographic Photoplates

PAUL P. BEY AND JAMES G. ALLARD

*Optical Materials Branch
Optical Physics Division*

April 8, 1968



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REF ID: A670593
JUN 19 1968
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NAVAL RESEARCH LABORATORY
Washington, D.C.

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ABSTRACT

To determine impurity concentrations in solids using mass spectrographs, ion-sensitive emulsions are used. The principal problem in obtaining quantitative results from a mass spectrum recorded on a photographic plate is determining the characteristic response curve. Calculations required to determine the parameters in an empirical function which accurately represents the response curve of Ilford Q2 emulsions are programmed in FORTRAN language. The program input data consists of the impurity-ion mass, line density, and exposure; the impurity-ion concentration and the detection limit are calculated and printed as output.

PROBLEM STATUS

This is a final report on the computer program for determining impurity concentrations in solids.

AUTHORIZATION

NRL Problem P03-07A
Project ARPA Order Number 418

Manuscript submitted October 4, 1967.

A COMPUTER PROGRAM FOR THE QUANTITATIVE INTERPRETATION OF MASS SPECTROGRAPHIC PHOTOPLATES

INTRODUCTION

Solid-state mass spectrographs normally use ion-sensitive photographic plates as the output detector, due to the wide variation in ion intensity of the rf vacuum spark source. In addition, the photoplate is an excellent ion integrator and has the ability to detect and to record a wide range of masses simultaneously.

There are two principal procedures used for the analytical interpretation of photographic plates: (a) visual inspection (1), where an experienced analyst visually compares line densities and calculates concentrations from relative exposures, and (b) the photometric method (1-3), where a microdensitometer is used to determine line densities and the data are processed by manual calculations. The first method produces semiquantitative results; the latter method, by eliminating errors arising from visually matching densities, line width, emulsion background, etc., can produce quantitative results.

This report is not intended to argue in favor of one method over the other. Both methods are useful. However, if the densitometric method is used, the manual labor required to process the data is so great that the routine computation can be done best by a digital computer.

The primary problem in obtaining a quantitative analysis of a mass spectrum recorded on a photoplate is determining the characteristic response curve of the emulsion. There are several "functionless" methods (4) for obtaining the characteristic response curve, but the method most widely associated with mass spectrography is the modification by Duke (2), based on the "two-line technique" described by Churchill (5) for use in optical spectrography. The characteristic response curve may also be obtained by a "functional" method. An empirical function given by Huli (6) accurately represents the entire response range of Ilford Q2 emulsions, thus permitting all analytical data to be evaluated.

Both the functionless and the functional method have individual merit. The Churchill two-line method is applicable only for elements possessing an appropriate isotopic distribution, and it requires many pairs of measurements of two lines having a known intensity ratio (such as isotopes whose abundance ratio falls within the requisite range). In addition, only information from a single element is used to construct the characteristic curve, but Owens and Giardino (7) have demonstrated that emulsion response exhibits ion-mass dependence, ion-energy dependence, and, possibly, chemical dependence. However, Kennicott (8) has described a computer program using this method.

The functional method, in which the data are fitted to an empirical curve, is easier to program. However, it tends to force the data into a predetermined formula, and any errors of the ion-beam integrator are not corrected. Woolston (9) has described a computer program written in assembly-system language, using the functional method.

The program described below is modeled after Woolston's program, but it is written in FORTRAN IV, because NRL's CDC 3800 computer is more receptive to FORTRAN language. Generally the mathematical functions and symbols of both Woolston's program and our program are the same; there are slight additions or deletions that suited our

personal approach. However, the parameters calculated from the same data are essentially the same.

MATHEMATICAL FORMULATION

The equation for the photographic calibration curve given by Hull (6) may be expressed by

$$K_x a_{x,i} E_x = \left(\frac{100 - T_{L_i}}{T_{L_i} - T_{SAT}} \right)^{1/R_x} \quad (1)$$

where, for a sample component x ,

- K_x is directly proportional to the component concentration in the total ion beam striking the plate and to the photoplate sensitivity,
- $a_{x,i}$ is the abundance of isotope i of the sample component,
- E_x is the exposure, indicated by the beam-monitor integrator, in nanocoulombs,
- T_L is the percent line transmission (corrected for background) of the spectral line i ,
- T_{SAT} is the percent transmission of the spectral line i for an infinite (saturation) exposure, and
- R_x is proportional to the maximum slope of the photographic response curve.

The corrected line transmission T_L in Eq. (1) is given by

$$T_L = \frac{100 + T_{SAT} \left[\left(\frac{100 - T_{LB}}{T_{LB} - T_{SAT}} \right)^{1/R} - \left(\frac{100 - T_B}{T_B - T_{SAT}} \right)^{1/R} \right]^R}{1 + \left[\left(\frac{100 - T_{LB}}{T_{LB} - T_{SAT}} \right)^{1/R} - \left(\frac{100 - T_B}{T_B - T_{SAT}} \right)^{1/R} \right]^R} \quad (2)$$

where T_{LB} is the measured line transmission and T_B is the measured background transmission, both in percent transmission.

The sensitivity of the emulsion is proportional to $M^{0.6}$, where M is the mass of the ion. When this factor is included the equation for calculating the impurity-ion concentration C_i in ppma is determined from Eq. (1) and becomes

$$C_i = \frac{10^6 (MF_x)^{0.6} S_r K_x C_r}{(MF_r)^{0.6} S_x K_r} \quad (3)$$

where MF is a factor proportional to the mass in atomic mass units, C_r is the fractional concentration of the reference ion, and S is the relative sensitivity coefficient (unless both S_x and S_r are known, they are taken to be unity).

When the lines have finite width the concentrations are corrected by a factor equal to the line width. Thus, Eq. (3) must be multiplied by $w_{x,i} / w_{r,j}$, where w is the width of

the line as determined at the points of half-maximum on the intensity profile. When the background is considered, these points correspond to the transmission

$$T_{Lw} = \frac{100 + T_{SAT} \left[\frac{1}{2} \left(\frac{100 - T_{LB}}{T_{LB} - T_{SAT}} \right)^{1/R_z} + \frac{1}{2} \left(\frac{100 - T_B}{T_B - T_{SAT}} \right)^{1/R_z} \right]^{R_z}}{1 + \left[\frac{1}{2} \left(\frac{100 - T_{LB}}{T_{LB} - T_{SAT}} \right)^{1/R_z} + \frac{1}{2} \left(\frac{100 - T_B}{T_B - T_{SAT}} \right)^{1/R_z} \right]^{R_z}} \quad (4)$$

NUMERICAL CALCULATIONS

When only one data point is available, R_z is taken to be 1. For two data points (1 and 2) R_z is determined from the relationship

$$R_z = \frac{\log \left[\left(\frac{100 - T_{L1}}{T_{L1} - T_{SAT}} \right) \left(\frac{T_{L2} - T_{SAT}}{100 - T_{L2}} \right) \right]}{\log \left(\frac{a_1 E_1}{a_2 E_2} \right)} \quad (5)$$

which may be derived from Eq. (1). Only three data points are allowed in the program. When three points are used, an average value of R_z (equal to the root-mean-square of R_z obtained from the calculation for the three combinations of pairs of data points) is calculated and is designated as R_{AV} . If R_z is either greater than 1.25 or less than 1, R_{AV} is set equal to 1.0. Upon substituting R_{AV} into Eq. (1), numerical values of K_z are calculated for each data point, and the root mean square of this result, denoted by K_{AV} , is substituted into Eq. (3) to determine the ion concentration.

Numerical values of T_{Lw} are calculated from Eq. (4) for each data point; an average value of the correction for line width can be determined by the densitometric measurements, and the factor $w_{x,i}/w_{r,i}$ can be applied to Eq. (3).

The detection limit is determined by using R_{AV} to calculate K_z for the maximum component exposure, setting $T_L = 100\%$ and $T_B = 98\%$ in Eq. (1) and substituting these values of K_z in Eq. (3).

GENERAL DESCRIPTION OF THE PROGRAM

Expressions are programmed for the calculations described in the previous section for experimental data read in from punched cards. Tables of isotope abundances and identifications are punched on IBM cards and are read in preceding the experimental data cards. If an incorrect isotope identification is made on the corresponding data card, NO ISOTOPE LISTED is printed, and the calculation for that isotope is bypassed. Other errors are determined by the system error identifiers. Formats of the experimental data cards are given in Table 1.

The input data are punched on cards. A table of isotope abundances is read in with the measured data. Table 2 gives a definition of the symbols used in the program.

Cards 3 through $4 + (N - 1)$ are included, in order, for each isotope. Three cards (identified as 23-1, 23-2, and 23-3) may be inserted into the program between cards 23 and 24. The data are then punched in as optical density.

Table 1
Input Data Format

Data Card Number	Format	Symbols
1	(55H)	Run Identification
2	(1x, I4, 10x, E11.4, 2x, F10.4)	NO RUNS, REF CONC, EIM X
3	(1x, F4.1, 9x, F7.2, 10x, I2, 10x, A8, A1, 7x, F5.2)	CHG, ATWT, N, Q, S*
4 through 4 + (N - 1)	(A8 1x, F8.4, 2 (2x, F8.4), 3x, E10.3)	ID, TLB, TB, TSAT, EI

*The sequence of cards 3 through 4 + (N - 1) is repeated a number of times equal to the number of runs.

Table 2
Definition of Symbols

Card Number	Symbol	Definition
2	NO RUNS	The number of isotopes processed
2	Ref Conc	The concentration of the reference isotope
2	EIMAX	The maximum photoplate exposure in nanocoulombs
3	CHG	The isotopic charge
3	ATWT	The average atomic weight of the element
3	N	The number of data points used for the isotope processed
3	Q	The comments
3	S	(Defined in text)
4	ID	The atomic symbol and mass number
4	TLB, TB, TSAT, EI	(Defined in text)

The first 1-2 characters in the A8 specification of ID refers to the atomic symbol, and the last 1-3 characters correspond to the atomic mass.

ACKNOWLEDGMENT

Mr. Kenneth Moran of the NRL Computer Center is gratefully acknowledged for his programming assistance.

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Appendix A PROGRAM LISTING

PROGRAM SPECTRA	1
DIMENSION TLB(3),TB(3),TSAT(3),TLW(3),TL(3),R(3),KX(3),ID2(3),	2
CID(3),AB(287),IDEN(287),MASS(287),MI(3),EI(3),AI(3),Q(2)	3
TYPE REAL MFACTOR,KAV,KX,MI,XDL,KAVR	4
201 FORMAT(7F10.5)	5
202 FORMAT(9A8)	6
203 FORMAT(19I4)	7
204 FORMAT(2(0I6))	8
READ201,AB	9
READ202,IDEN	10
READ203,MASS	11
READ204,MSK1,MSK2	12
LINES=47	13
READ 206	14
PRINT 205	15
PRINT 206	16
CALL HEADING	17
RFAD30 ,NO RUNS,REF CONC,EIMAX	18
DO 26 I=1,NO RUNS	19
READ 31,CHG,AT WT,N,Q,S	20
MFACTOR=(AT WT/(30.0*CHG))*C.6	21
DO 4 J=1,N	22
READ 32,ID(J),TLB(J),TB(J),TSAT(J),EI(J)	23
TLB(J)=10.0*(-TLB(J))*100.0	23-1
TB(J)=10.0*(-TB(J))*100.0	23-2
TSAT(J)=10.0*(-TSAT(J))*100.0	23-3
ID2(J)=MSK2.AND.ID(J)	24
L=0	25
1 L=L+1	26
IF(L.GT.287)33,2	27
2 IF(ID(J).EQ.IDEN(L))3,1	28
3 AI(J)=AB(L)/100.0	29
MI(J)=MASS(L)	30
CALL CALC TL(TLB(J),TB(J),TSAT(J),TL(J))	31
4 CONTINUE	32
ID1=MSK1.AND.ID	33
IF(N.EQ.1)5,6	34
5 R=1.0	35
R(2)=1.0	36
RAV=1.0	37
CALL CALC K(R,TL,TSAT,EI,AI,KX)	38
CALL CALC TLW(R,TLB,TB,TSAT,TLW)	39
KAV=MFACTOR*KX	40
GO TO 11	41
6 IF(N.EQ.2)7,9	42
7 CALL CALC R(TL,TL(2),AI*EI,AI(2)*EI(2),TSAT,TSAT(2),R)	43
R(2)=R	44
RAV=R	45
DO 8 M=1,2	46
CALL CALC K(R,TL(M),TSAT(M),EI(M),AI(M),KX(M))	47
8 CALL CALC TLW(R,TLB(M),TB(M),TSAT(M),TLW(M))	48
KAV= SQRTF(KX*KX(2))*MFACTOR	49
GO TO 11	50
9 DO 10 M=2,3	51

10 CALL CALC R(TL,TL(M),AI*EI,AI(M)*EI(M),TSAT,TSAT(M),R(M-1))	52
CALL CALC R(TL(2),TL(3),AI(2)*EI(2),AI(3)*EI(3),TSAT(2),TSAT(3),	53
CR(3))	54
EX=1.0/3.0	55
RAV=(R*R(2)*R(3))**EX	56
DO 101 M=1,3	57
CALL CALC K(RAV,TL(M),ISAT(M),EI(M),AI(M),KX(M))	58
101 CALL CALC TLW(RAV,TLB(M),TB(M),TSAT(M),TLW(M))	59
KAV=(KX*KX(2)*KX(3))**EX*MFACTOR	60
11 IF(I.EQ.1)12,13	61
12 CI=REF CONC	62
SR=S	63
KAVR=KAV	64
GO TO 131	65
13 CI= SR*KAV/(S*KAVR)* REF CONC	66
131 IF(N.EQ.1)14,15	67
14 M1=1	68
GO TO 20	69
15 N1=N-1	70
DO 18 M=1,N1	71
IF(AI(M).LT.AI(M+1))16,17	72
16 M1=M+1	73
GO TO 18	74
17 M1=M	75
18 CONTINUE	76
IF(N.EQ.3)19,20	77
19 RX=RAV	78
GO TO 21	79
20 RX=R	80
21 CALL CALC K(RX,98.0,0.0,0.0,EIMAX,AI(M1),KDL)	81
DET LIM=SR*KDL/(KAVR*S)* REF CONC* MFACTOR	82
PRINT 27,1D1,CI,DET LIM,Q(1),Q(2),1D2(1),TLB(1),TB(1),TSAT(1),	83
CTL(1),TLW(1),R(1),KX(1),MFACTOR,S,EI(1)	84
IF(N.EQ.1)24,23	85
23 DO 231 LN=2,N	86
PRINT 28,1D2(LN),TLB(LN),TB(LN),TSAT(LN),TL(LN),TLW(LN),R(LN),	87
CKX(LN),EI(LN)	88
231 CONTINUE	89
24 PRINT 29,RAV,KAV	90
LINES=LINES-N-3	91
IF(LINES.LT.0)25,26	92
25 LINES=47	93
PRINT 205	94
PRINT 206	95
CALL HEADING	96
26 CONTINUE	97
GO TO 35	98
33 PRINT 34	99
34 FORMAT(19H ISOTOPE NOT LISTED)	100
IF(I.EQ.NO RUNS)35,36	101
27 FORMAT(3X,A2,4X,E10.3,2X,F8.4,3X,A3,A1,2X,R3,5(2X,F5.1),5X,F6.3,	102
C6X,E10.3,2X,F6.3,2X,F5.2,2X,E8.1)	103
28 FORMAT(43X,R3,5(2X,F5.1),5X,F6.3,6X,E10.3,17X,E8.1)	104
29 FORMAT(82X,4HRAV=,F6.3,2X,4HKAV=,E10.3,/))	105
205 FORMAT(1H1)	106
206 FORMAT(55H)	107
30 FORMAT(1X,14,10X,E11.4,2X,F10.4)	108
31 FORMAT(1X,F4.1,9X,F7.2,1CX,12,10X,A8,A1,7X,F5.2)	109
32 FOPMAT(A8,1X,F8.4,2(2X,F8.4),3X,E10.3)	110
36 IF(J.EQ.N)26,37	111
37 JE1=J+1	112
DO 38 JE=JE1,N	113
READ 32,1D(JE),TLB(JE),TB(JE),ISAT(JE),EI(JE)	114

38	CONTINUE	115
	GO TO 6	116
35	CONTINUE	117
	END	118
	SUBROUTINE HEADING	119
	PRINT 1	120
1	FORMAT(/1X,6HSYMBOL,6X,3HPPM,5X,9HDET LIMIT,3X,8HCOMMENTS,2X,	121
	4HMASS,3X,3HILB,4X,2HFB,5X,4HISAT,4X,2HTL,4X,3HTLW,8X,1HR,13X,	122
	CIHK,9X,2HMF,6X,1HS,8X,2HEI/)	123
	END	124
	SUBROUTINE CALC K(R,TL,TSAT,EI,AI,KX)	125
	TYPE REAL KX	126
	IF(R.GT.1.25)1,2	127
1	RX1=1.0/1.25	128
	GO TO 5	129
2	IF(R.LT.1.0)3,4	130
3	RX1=1.0	131
	GO TO 5	132
4	RX1=1.0/R	133
5	KX=((100.0-TL)/(TL-TSAT))**RX1/(AI*EI)	134
	END	135
	SUBROUTINE CALC TLW(R,TLB,TB,TSAT,ILW)	136
	IF(R.GT.1.25)1,2	137
1	RX=1.25	138
	RX1=1/1.25	139
	GO TO 3	140
2	RX=R	141
	RX1=1.0/R	142
3	TS1=((100.0-TLB)/(TLB-TSAT))**RX1/2.0	143
	TS2=((100.0-TB)/(TB-TSAT))**RX1/2.0	144
	TS3=(TS1+TS2)**RX	145
	TLW=(100.0+TSAT*TS3)/(1.0+TS3)	146
	END	147
	SUBROUTINE CALC TL(TLB,TB,TSAT,TL)	148
	TS=((100.0-TLB)/(TLB-TSAT)-(100.0-TB)/(TB-TSAT)	149
	TL=(100.0+TSAT*TS)/(1.0+TS)	150
	END	151
	SUBROUTINE CALC R(TL1,TL2,AE1,AE2,TSAT1,TSAT2,R)	152
	R=LOGF((100.0-TL1)/(TL1-TSAT1)*(TL2-TSAT2)/(100.0-TL2))/	153
	CLOGF(AE1/AE2)	154
	END	155

Appendix B
SAMPLE PROGRAM OUTPUT

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SAMPLE PROGRAM 5-508 KER:KCL 8-10-67

SYMBOL	PPM	DET LIMIT	COMMENTS	MASS	TLB	TB	TSAT	TL	TLW	R	K	MP	S	EI
K	5.000+005	0.0705	REFERENCE	41	56.5	94.0	2.6	50.2	72.1	1.115	3.701+003	1.172	1.00	3.0+003
				41	27.1	91.2	2.6	27.7	43.1	1.171	3.509+003			1.0+002
				41	10.5	83.8	2.6	10.6	16.3	1.232	3.775+003			3.0+002
									RAV= 1.172		4.346+003			
CL	5.042+003	0.0052		35	53.5	97.3	2.6	54.2	70.1	1.120	3.905+001	1.105	1.00	3.0+002
				35	24.5	96.4	2.6	24.7	39.0	1.120	3.905+001			1.0+001
E	1.644+001	0.0140		10	94.0	96.6	2.6	90.1	95.7	1.308	2.321+003	0.542	1.90	1.0+002
				10	56.5	76.2	2.6	66.2	65.3	1.308	2.994+003			1.0+003
NA	5.912+001	0.0020		23	78.7	99.3	2.6	79.1	81.6	0.577	9.094+001	0.052	1.00	3.0+001
				23	65.3	99.3	2.6	65.6	73.6	0.577	5.483+001			1.0+000
B	6.004+002	0.0034		11	94.2	96.8	2.6	97.2	95.5	1.550	7.399+004	0.542	1.00	1.0+002
				11	43.9	77.3	2.6	50.0	56.0	1.550	1.286+003			1.0+003
FE	1.109+000	0.0001		56	58.3	81.3	2.6	67.1	68.5	1.372	6.362+003	1.452	1.00	1.0+002
				56	9.1	35.0	2.6	10.1	14.1	1.372	7.940+003			1.0+003
CU	0.097+001	0.0116		63	75.9	90.8	2.6	62.1	83.0	1.273	4.390+003	1.569	1.00	1.0+002
				63	17.9	47.5	2.6	21.3	26.2	1.273	4.502+003			1.0+003
SI	3.328+000	0.1041		29	34.3	72.4	2.6	38.9	47.5	1.659	3.219+002	0.961	1.00	1.0+003
				30	45.2	70.3	2.6	55.3	55.5	1.659	2.813+002			1.0+003
MM	2.925+001	0.0034		55	23.3	35.8	2.6	37.8	28.1	1.000	1.768+003	1.430	1.00	1.0+003
									RAV= 1.000		2.542+003			
RB	2.166+000	0.0061	F ASSUMED	85	50.5	76.9	2.6	59.1	60.8	1.000	1.004+002	1.874	1.00	1.0+002
									RAV= 1.000		1.003+002			

Appendix C
DATA CODING FORM

UNCLASSIFIED

Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Naval Research Laboratory Washington, D. C. 20390		2a. REPORT SECURITY CLASSIFICATION Unclassified	
		2b. GROUP	
3. REPORT TITLE A COMPUTER PROGRAM FOR THE QUANTITATIVE INTERPRETATION OF MASS SPECTROGRAPHIC PHOTOPLATES			
4. DESCRIPTIVE NOTES (Type of report and inclusive dates) This is a final report on the computer program for determining impurity concentration in solids.			
5. AUTHOR(S) (First name, middle initial, last name) Paul P. Bey and James G. Allard			
6. REPORT DATE April 8, 1968		7a. TOTAL NO. OF PAGES 16	7b. NO. OF REFS 9
8a. CONTRACT OR GRANT NO. NRL Problem P03-07A		8b. ORIGINATOR'S REPORT NUMBER(S) NRL Report 6655	
b. PROJECT NO. ARPA Order Number 418			
c.		8d. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	
d.			
10. DISTRIBUTION STATEMENT This document has been approved for public release and sale; its distribution is unlimited.			
11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY Advanced Research Projects Agency Washington, D. C. 20301	
13. ABSTRACT <p>To determine impurity concentrations in solids using mass spectrophotographs, ion-sensitive emulsions are used. The principal problem in obtaining quantitative results from a mass spectrum recorded on a photographic plate is determining the characteristic response curve. Calculations required to determine the parameters in an empirical function which accurately represents the response curve of Ilford Q2 emulsions are programmed in FORTRAN language. The program input data consists of the impurity-ion mass, line density, and exposure; the impurity-ion concentration and the detection limit are calculated and printed as output.</p>			

KEY WORDS

LINK A

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LINK C

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Mass spectrographic computer program
Impurity analysis
Mass spectrographic data processing
Analysis of solids